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Microstructural investigation, using small-angle neutron scattering (SANS), of Optifer steel after low dose neutron irradiation and subsequent high temperature tempering

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Abstract

The microstructural effect of low dose neutron irradiation and subsequent high temperature tempering in the reduced activation ferritic/martensitic steel Optifer (9.3 Cr, 0.1 C, 0.50 Mn, 0.26 V, 0.96 W, 0.66 Ta, Fe bal wt%) has been studied using small-angle neutron scattering (SANS). The investigated Optifer samples had been neutron irradiated, at 250 °C, to dose levels of 0.8 dpa and 2.4 dpa. Some of them underwent 2 h tempering at 770 °C after the irradiation. The SANS measurements were carried out at the D22 instrument of the High Flux Reactor at the Institut Max von Laue – Paul Langevin, Grenoble, France. The differences observed in nuclear and magnetic SANS cross-sections after subtraction of the reference sample from the irradiated one suggest that the irradiation and the subsequent post-irradiation tempering produce the growth of non-magnetic defects, tentatively identified as microvoids.

1. Introduction

Under neutron irradiation complex microstructural phenomena occur in ferritic/martensitic steels for future fusion reactors and for accelerator driven systems (ADS), implying changes in precipitate composition and the growth of helium bubbles and microvoids, with consequent changes in the thermo-mechanical properties [1–3]. Such steels must therefore be optimised to improve the resistance to swelling and helium effects and their microstructural stability under high temperature irradiation. The microstructural phenomena underlying the changes under irradiation in the ductile-to-brittle transition temperature (DBTT) [4,5] require a careful investigation: techniques such as transmission electron microscopy (TEM) and small-angle neutron scattering (SANS) are quite useful in this regard. SANS allows to distinguish non-magnetic defects, such as microvoids or helium bubbles, and magnetic ones, such as certain kinds of precipitates;

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additional and unique information on defect composition can be obtained using polarised SANS [6–9]. This contribution presents new results of a SANS study carried out to investigate the microstructural effect of neutron irradiation and subsequent tempering in a reduced activation ferritic/martensitic steel of interest for fusion technology.

2. Material characterization

The Optifer steel was investigated, having the following chemical composition: 9.3 Cr, 0.1 C, 0.50 Mn, 0.26 V, 0.96 W, 0.066 Ta Fe bal (wt%) [10–12]. Both the irradiated and the reference samples had been submitted to the standard metallurgical treatment (950 °C for 30 min then 750 °C for 2 h).

The samples were irradiated by exposure to thermal neutrons at the High Flux Reactor – Petten up to dose levels of 0.8 and 2.4 dpa (displacement per atom) at 250 °C. Some of these samples were tempered 2 h at 770 °C after the irradiation. Unirradiated reference samples, submitted to the same thermal treatments as the irradiated ones, were also prepared. The size of both the irradiated and unirradiated specimens utilized for the SANS experiments was $4 \times 10\ mm^2$ in surface and 1 mm in thickness. The results of TEM observations of these Optifer samples [11] show that after irradiation α′-precipitates (Cr-rich phase), dislocation loops, microvoids and helium bubbles are locally present between the lath boundaries, which appear decorated with carbides such as $(Fe, Cr, V, W)_{23}C_6$ or TaC, in different contents depending on the irradiation conditions and on the composition of the material. However it is difficult to obtain quantitative information from such local observations, limited both in statistics and because of the TEM resolution in such magnetic materials.

3. Experimental technique

Reference is made to [13,14] and to previous works [6–9] for a general presentation of SANS and of its application to the study of martensitic steels. SANS measurements were carried out using the D22 diffractometer at the Institut Max von Laue – Paul Langevin (ILL), in Grenoble. In a first series of SANS measurements, using a polarised neutron beam, the 0.8 dpa irradiated Optifer samples were investigated, obtaining the results reported in [6,8]. The experimental conditions defined for investigating the 2.4 dpa irradiated Optifer samples, with unpolarised neutron beam, were a neutron wavelength λ of 6 Å and a sample-to-detector distance of 2 m. Defining the scattering vector $Q = 4\pi \sin\theta/\lambda$, where 2θ is the scattering angle and λ the neutron wavelength, Q values ranging between 0.03 $Å^{-1}$ and 0.2 $Å^{-1}$ were obtained, corresponding in the real space to particle sizes between approximately 10 and 100 Å. After correction for background noise, detector efficiency, and attenuation factor the SANS cross-section in physical units ($cm^{-2}\ sterad^{-1}$) was obtained by a calibration of the neutron flux, measuring water in a quartz cell, and by means of the ILL standard programs [15]. The SANS cross-section of each reference samples was subtracted from the SANS cross-section of the corresponding irradiated sample, in order to distinguish as accurately as possible the effect of the irradiation itself from all the other microstructural effects arising during the heating at 250 °C for a duration corresponding to the irradiation time.

In the case of magnetic samples, the total SANS cross-section $d\Sigma(Q)/d\Omega$ (where Ω stands for the solid angle) can be written as

$$d\Sigma(Q)/d\Omega = d\Sigma(Q)/d\Omega_{nucl} + d\Sigma(Q)/d\Omega_{mag} \sin^2 \alpha, \tag{1}$$

where $d\Sigma(Q)/d\Omega_{nucl}$ and $d\Sigma(Q)/d\Omega_{mag}$ are the nuclear and magnetic SANS cross-sections respectively and α is the azimuthal angle on the detector plane. A saturating horizontal magnetic field of 1 T was applied perpendicular to the incoming neutron beam in order to distinguish $d\Sigma(Q)/d\Omega_{nucl}$ and $d\Sigma(Q)/d\Omega_{mag}$, so that only nuclear scattering occurs in the horizontal plane, while nuclear and magnetic scattering occur in the vertical one (the purely magnetic scattering is obtained as the difference between the vertical and horizontal SANS cross-sections).

The nuclear plus magnetic to nuclear cross-sections ratio $R(Q)$ (also defined as 'A' in the literature) can be written as

$$\begin{aligned} R(Q) &= (d\Sigma(Q)/d\Omega_{nucl} + d\Sigma(Q)/d\Omega_{mag})/d\Sigma(Q)/d\Omega_{nucl} \\ &= 1 + (\Delta\rho)^2_m (\Delta\rho)^2_n, \end{aligned} \tag{2}$$

where $(\Delta\rho)^2_m$ and $(\Delta\rho)^2_n$ are respectively the neutron magnetic and nuclear scattering length density square differences between matrix and the microstructural inhomogeneities giving rise to the observed SANS effect to [13,14]. In complex steels, such as Optifer, inhomogeneities with different